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14. ABSTRACT

In this effort, significant emphasis was placed on the epitaxial growth and in situ doping of ZnO for optoelectronic devices. The materials research efforts on ZnO have resulted in device-quality material on sapphire substrates for the desired laser diodes, where the eventual material quality improvements necessary for the devices are anticipated for future growth on ZnO substrates. P-type doping of ZnO was demonstrated using nitrogen, though the p-type behavior was found to be unstable over time. In situ arsenic and antimony doping of ZnO was demonstrated, exhibiting either weak p-type behavior or mixed p-type/n-type conduction, though with stable behavior over time. In addition to the binary ZnO, the growth of MgZnO and ZnO/MgZnO heterojunctions was studied. Quantum wells exhibiting efficient radiative recombination were demonstrated, and may form the basis of future ultraviolet LEDs and lasers. Device applications of the ZnO/MgZnO materials were studied, which included studies of ohmic contacts to ZnO, UV photoconductors, and thin film transistors. The integration of ferroelectric oxide thin films with ZnO was also investigated, as a potential means of locally inverting ZnO to p-type, and to achieve novel multi-functional devices for reconfigurable systems.

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Zinc oxide, ultraviolet light sources, semiconductor optoelectronic devices

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FINAL REPORT

Ultraviolet Electrically Injected Light Sources with Epitaxial Zno-Based Heterojunctions

Grant No: FA9550-04-1-0390

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OBJECTIVES

Ultraviolet semiconductor optoelectronic devices are of high importance for numerous applications including information processing and storage, solid-state lighting, UV spectroscopy, and solar blind detectors and imaging arrays, and transparent electronics for displays. These UV devices can also serve to meet Air Force objectives in areas covert tactical communications, high-speed information processing, solar blind sensing and imaging, and UV spectroscopy for biological and chemical sensing. Compound semiconductor materials based on ZnO and associated alloys are emerging as a promising candidate for UV optoelectronics. ZnO has a very large excitonic binding energy (60meV), suggesting the possibility of room temperature excitonic devices that may translate into ultra-low threshold lasers. ZnO also has lattice-matched alloys of MgZnO and CdZnO and an available high-quality native substrate of ZnO. In this work, the epitaxial growth, materials characterization, device fabrication and testing are studied to realize ZnO-based heterojunction ultraviolet sources. The primary objective of the project is the realization of a ZnO-based laser diode.

SUMMARY OF PROGRESS TOWARDS OBJECTIVES

In this effort, significant emphasis was placed on the epitaxial growth and in situ doping of ZnO for optoelectronic devices. The materials research efforts on ZnO have resulted in device-quality material on sapphire substrates for the desired laser diodes, where the eventual material quality improvements necessary for the devices are anticipated for future growth on ZnO substrates. P-type doping of ZnO was demonstrated using nitrogen, though the p-type behavior was found to be unstable over time. In situ arsenic and antimony doping of ZnO was demonstrated, exhibiting either weak p-type behavior or mixed p-type/n-type conduction, though with stable behavior over time. In addition to the binary ZnO, the growth of MgZnO and ZnO/MgZnO heterojunctions was studied. Quantum wells exhibiting efficient radiative recombination were demonstrated, and may form the basis of future ultraviolet LEDs and lasers. Device applications of the ZnO/MgZnO materials were studied, which included studies of ohmic contacts to ZnO, UV photoconductors, and thin film transistors. The integration of ferroelectric oxide thin films with ZnO was also investigated, as a potential means of locally inverting ZnO to p-type, and to achieve novel multi-functional devices for reconfigurable systems.

ACCOMPLISHMENTS

MBE growth of ZnO: As a starting point for this effort, a Riber 32 MBE system was modified to provide capability for oxide materials growth primarily through the addition of a turbomolecular pump and a microwave plasma source to provide atomic oxygen. The microwave plasma source is an electron cyclotron resonance (ECR) source at 2.45GHz, power up to 250W, from Oxford Scientific. Oxygen flow is controlled using a combination of a mass flow controller and a leak valve to provide a constant oxygen partial pressure during the MBE growth process. A schematic and photograph of the MBE system is shown in Figure 7. In addition, an oxygen resistant substrate heater and Zn effusion cell resistant to source oxidation have been installed to facilitate ZnO growth. The existing MBE system has demonstrated the ability to achieve high quality ZnO material. The epitaxial growth of ZnO on Al_2O_3 (0001) has been demonstrated, as illustrated by the RHEED pattern in Figure 2(a). Epilayers with smooth surface morphology have been obtained, as indicated by the 1μm x 1μm atomic force microscopy image in Figure 2(b), where a 4nm root mean square roughness was obtained. Double crystal x-ray diffraction measurements indicate high crystalline quality, as shown by the low full width at half maximum on the ZnO (0002) reflections in Figure 2(c). The materials also exhibit reasonable electronic properties, where room temperature Hall effect measurements indicate n-type behavior with carrier concentration and electron mobility in the range of $2x10^{17} - 2x10^{18}$ cm^{-3} and 10-80 $cm^2/V.s$, respectively. The ZnO materials possess good optical properties, where bandedge emission is observed in photoluminescence spectra as shown in Figure 2(d).

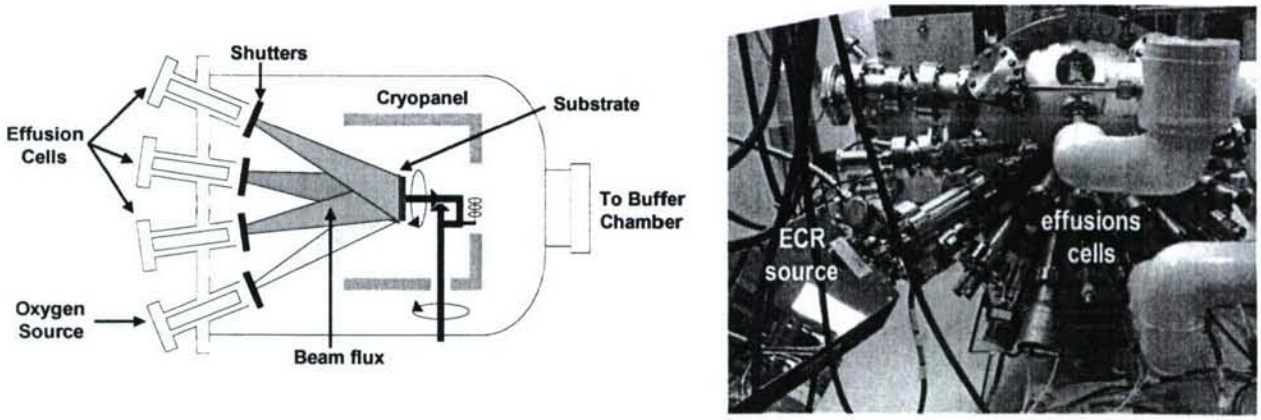


Figure 1: A schematic and rear view photograph of the existing oxide MBE system.

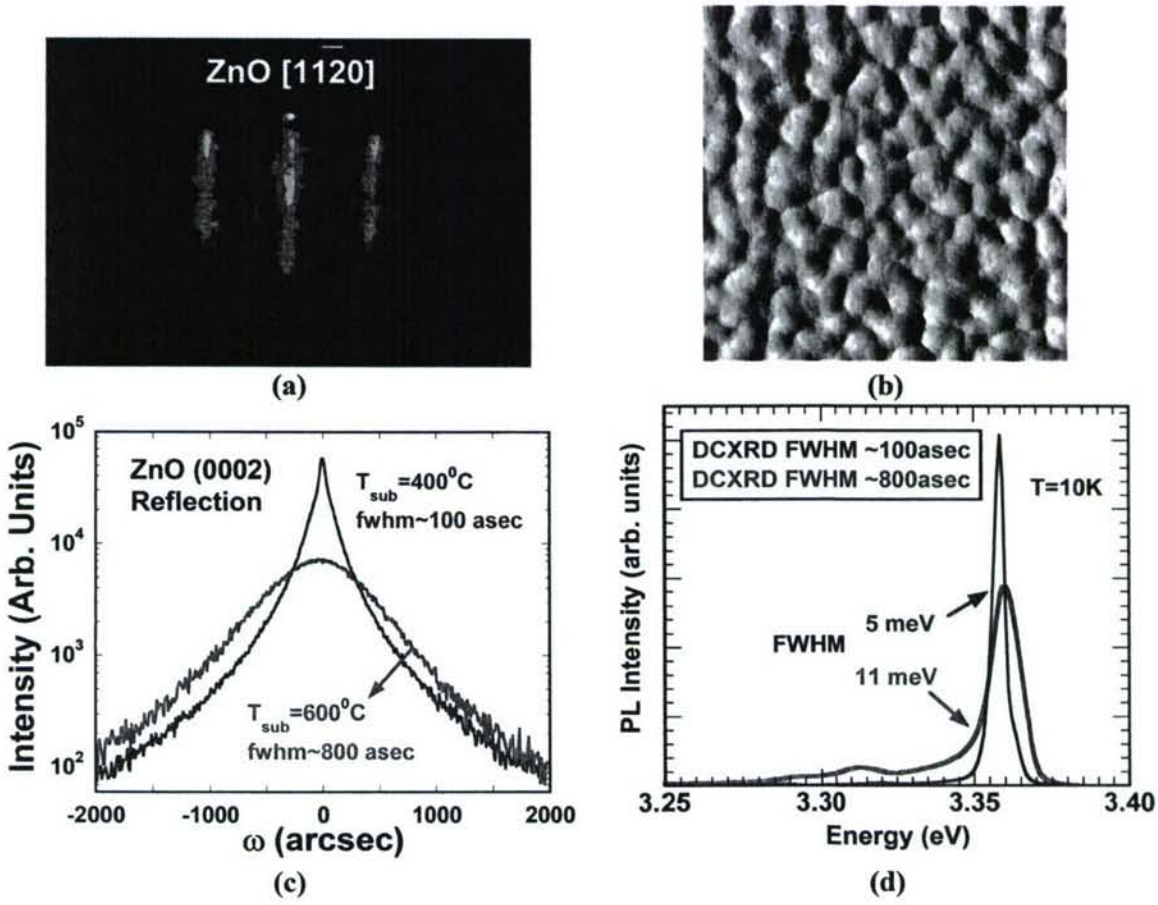


Figure 2: Characteristics of ZnO growth on Al_2O_3 (0001) given by (a) RHEED, (b) 1μm x 1μm atomic force microscopy image, (c) double crystal x-ray diffraction rocking curve of the (0002) diffraction, and (d) low temperature photoluminescence spectrum.

Growth and Optical Properties of ZnO/MgZnO Quantum Wells: The growth of $Mg_{0.15}Zn_{0.85}O$ and ZnO/ $Mg_{0.15}Zn_{0.85}O$ heterojunctions was studied during the course of this project. The usage of quantum wells and other lower dimensional structures associated with ZnO is expected to provide enhanced

radiative efficiency, so it is important to understand heterojunctions such as ZnO/MgZnO. The physics associated with ZnO heterojunctions may be particularly interesting due to the possibility of strong polarization effects, analogous to GaN and related materials. The growth and photoluminescence properties of single- and multiple-quantum wells were studied in this effort. Cross-sectional TEM images indicate heterojunctions with abrupt interfaces, as shown in Figure 3(a). The PL spectra of single quantum wells (SQW) demonstrate a clear blue shift with increased quantum confinement, for well widths ranging from 3-10nm (Figure 3(b)). A redshift of the PL peak energy for SQW with L_W>9.5nm in comparison to ZnO thin films indicate the existence of small built-in electric fields associated with polarization effects. The energetic position of the PL peak energy for all samples is consistent with some degree of polarization effects in the ZnO/MgZnO heterojunctions, based on calculation of transition energy given values for spontaneous and piezoelectric polarization reported in literature (P_{sp} values of -0.025 C/m^2 for ZnO and -0.072 C/m^2 for MgO; piezoelectric coefficients of e_{33}=1.34C/m^2 and e_{31}=0.57C/m^2). The small polarization effects observed in the quantum wells may be attributed to defects or potential fluctuations at the well/barrier interface.

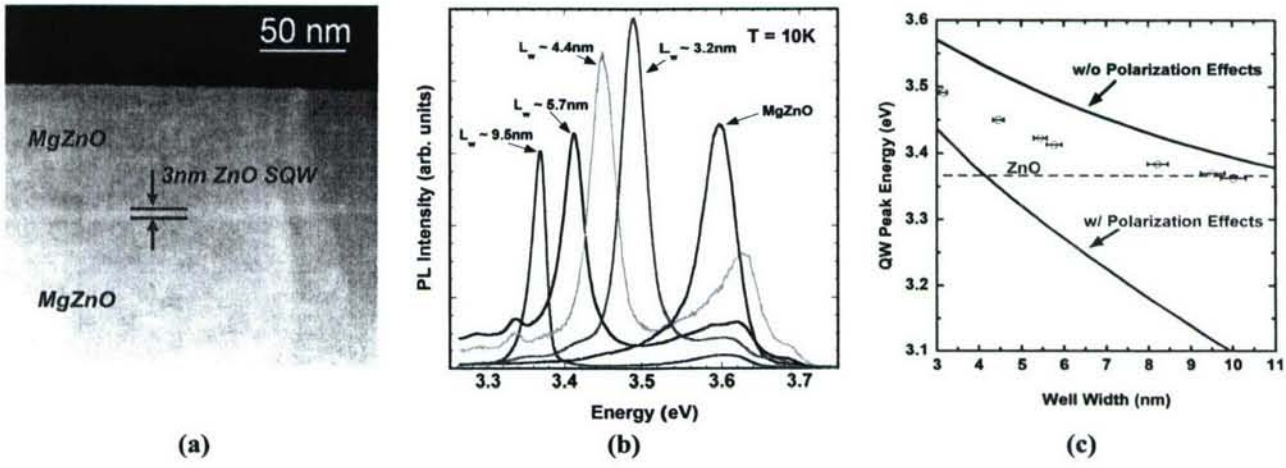


Figure 3: Characteristics of ZnO/$Mg_{0.15}Zn_{0.85}O$ single quantum wells showing (a) cross-sectional TEM image (b) peak shift of PL with varying well thickness and (c) comparison of PL peak shift with well thickness to calculated values with/without polarization effects in the heterojunction.

The temperature dependence of the quantum well PL characteristics (Figure 4) provided further information on the radiative properties. The quantum efficiency of a 5-layer MQW is comparable to or exceeding that of a high quality ZnO thin film, suggesting that the heterointerfaces do not strongly influence non-radiative recombination. Fitting the integrated PL intensity of the SQWs with a dual activation energy Arrhenius model revealed the existence of two non-radiative mechanisms. Enhanced quantum efficiency, decreased PL linewidth, and higher thermal activation energy was observed for narrower well width and increased quantum efficiency. The anomalous "S-shape" behavior observed in PL peak energy versus temperature is consistent with the thermal activation energy extracted for integrated PL intensity versus temperature, where an activation energy of approximately 4-5meV is obtained and attributed to the trapping of localized carriers/excitons at energy states associated with potential fluctuations at the ZnO/MgZnO interface. This work suggests that ZnO/MgZnO quantum wells with high optical quality may be obtained with enhanced properties associated with quantum confinement, and will serve to further guide efforts to understand the properties of ZnO/MgZnO heterojunctions.

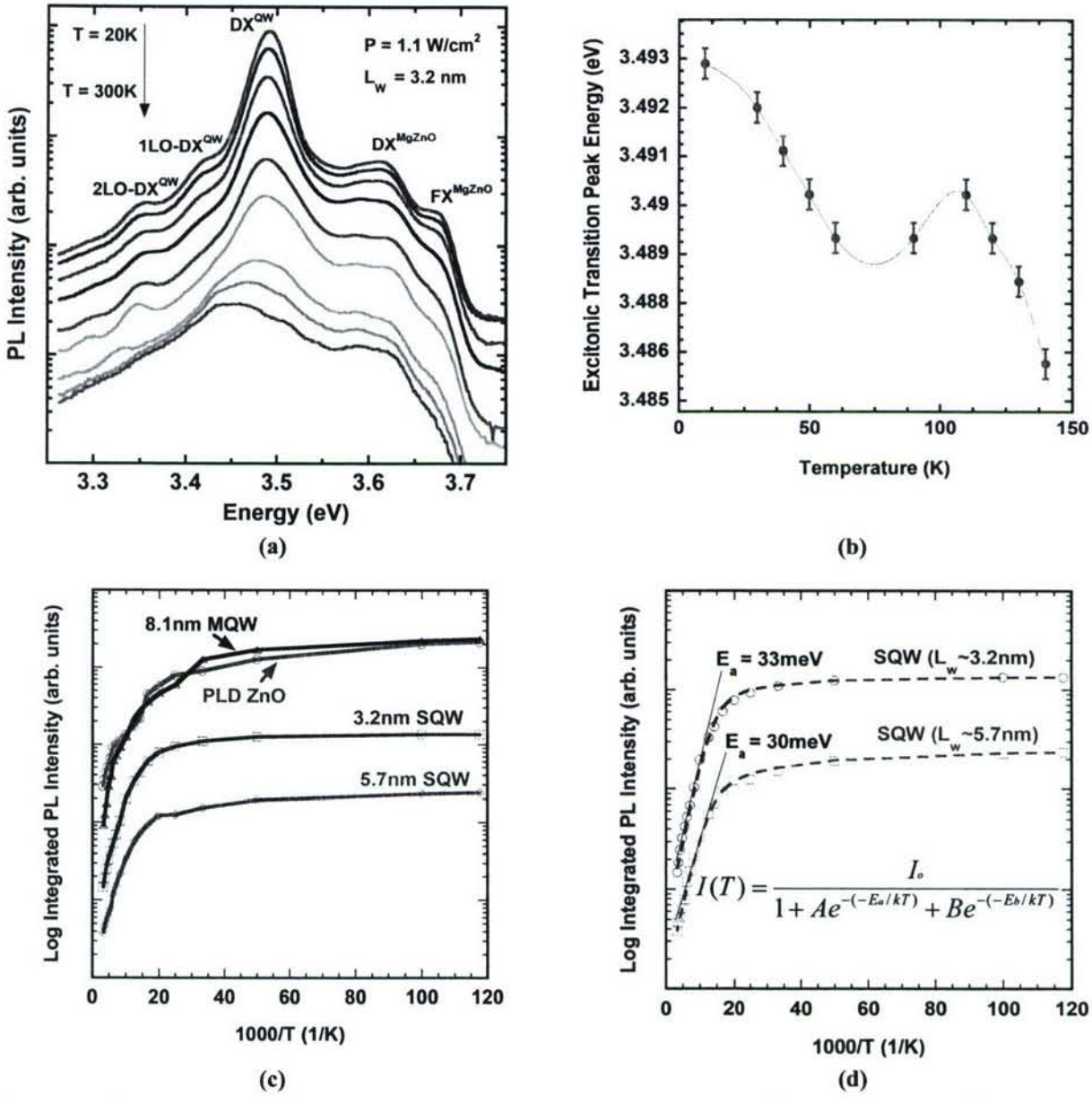


Figure 4: Temperature dependent photoluminescence properties of ZnO/MgZnO quantum wells showing (a) PL spectra at varying temperature (b) transition energy variation with temperature at low temperature exhibiting "s-shaped" behavior, (c) dependence of integrated intensity with temperature and (d) fit of integrated intensity temperature dependence with two non-radiative mechanisms.

In situ Doping of ZnO by MBE: Similar to other wide bandgap materials, one of the key challenges for ZnO emitters is the achievement of highly conductive p-type ZnO, requiring a stable shallow acceptor. A conventional approach for achieving p-type ZnO is a Group V substitutional impurity for oxygen. Nitrogen is the best match with respect to atomic radius, and provides the shallowest acceptor level. In year 1, we had demonstrated p-type ZnO experimentally using nitrogen doping ($p \sim 10^{18} cm^{-3}$). Photoluminescence spectra of nitrogen doped ZnO show a clear acceptor related transition, suggesting an acceptor energy of approximately 100meV (Figure 5(a)). This data agrees well with prior Hall effect and SIMS measurement data, where acceptor energy may be estimated through knowledge of nitrogen concentration and hole carrier concentration. Despite these accomplishments, nitrogen doping in ZnO has

been found to be unstable over time. We have in fact measured one of our nitrogen samples at several time periods, and have observed conversion from p-type to n-type behavior after approximately 60 days after initial growth. SIMS measurements show nitrogen concentration in the ZnO material after conversion to n-type behavior. The source for this instability is unclear and is debated by ZnO researchers. In year 2, we have been investigating arsenic doping, where the incorporation of an arsenic complex in ZnO is believed to provide shallow acceptor energy similar to nitrogen doping. We have demonstrated the in situ doping of ZnO with arsenic as shown by the SIMS data in Figure 6(a). Unfortunately, the arsenic doped ZnO material does not show conversion to p-type behavior. We have observed a clear effect of the compensation of native donors in ZnO with arsenic acceptors, as shown in Figure 6(b). The n-type carrier concentration shows a clear decrease (increased compensation) with increasing ratio of As/Zn ratio. We have also investigated doping of ZnO with antimony, with similar behavior of high antimony incorporation, but only weakly p-type material (~10^{16} cm^{-3}) or material with mixed p-type and n-type conduction. The samples exhibiting weak p-type behavior have very high resistivity, and not suitable for desired ZnO light emitters. Despite the high resistivity, arsenic and antimony doping demonstrate the potential to achieve stable p-type ZnO material.

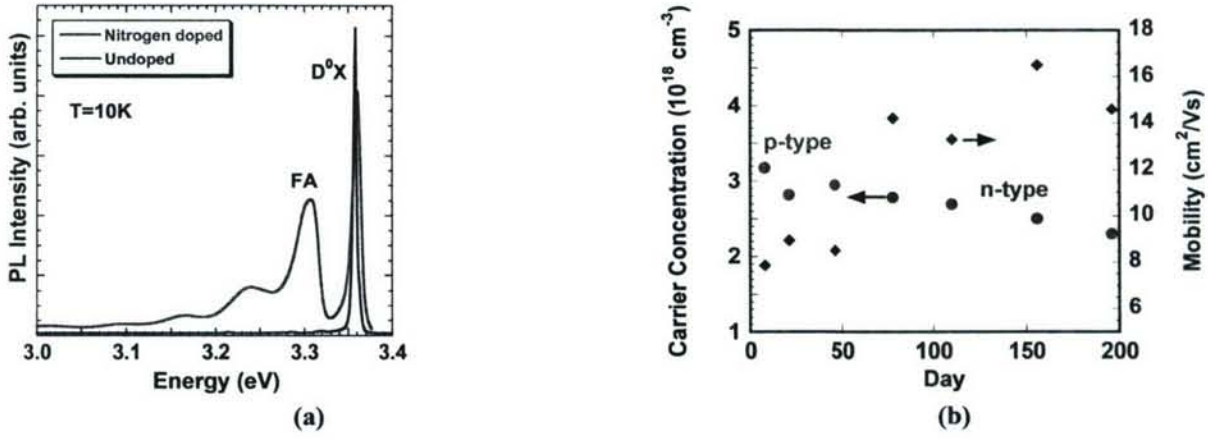


Figure 5: (a) Photoluminescence spectra for an undoped and nitrogen doped ZnO epilayer and (b) measured carrier concentration and mobility for a nitrogen doped sample demonstrating conversion to n-type behavior over time.

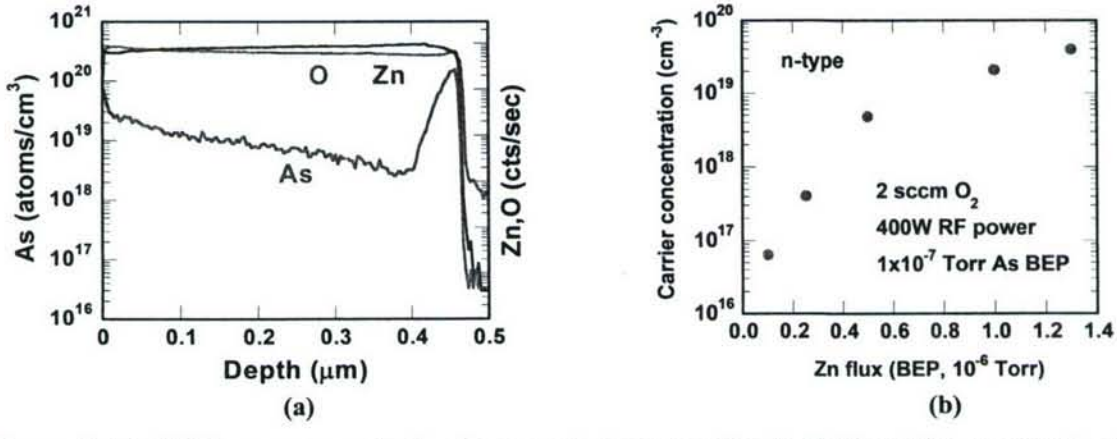
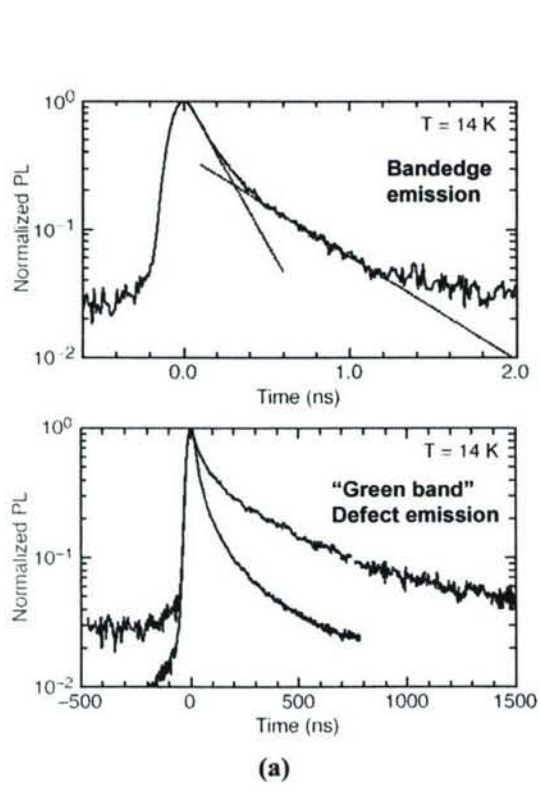
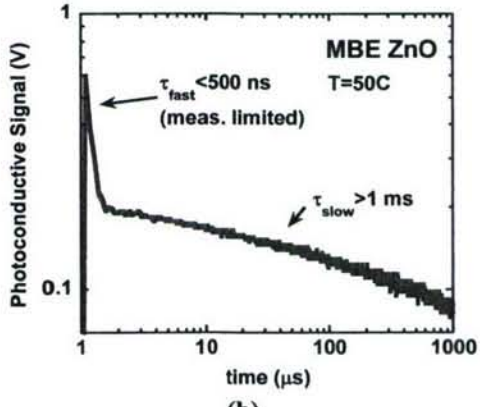


Figure 6: (a) SIMS measurements showing arsenic incorporation in ZnO and (b) measured n-type carrier concentration for ZnO doped with arsenic, demonstrating compensation of native donors with arsenic acceptors.

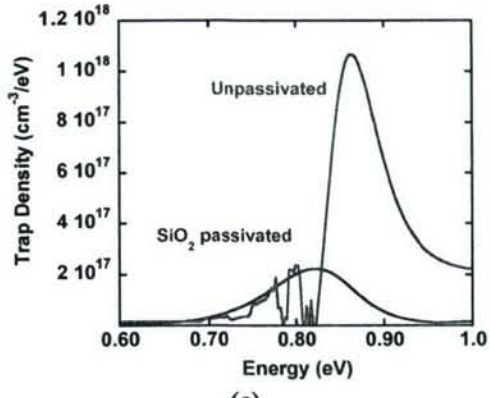
Defect studies in ZnO: Light emitting devices require a high radiative efficiency, and corresponding long non-radiative recombination rate. Radiative and non-radiative lifetimes in our ZnO epilayers have been investigated by time-resolved photoluminescence (in collaboration with A. Cartwright at the University at Buffalo) and photoconductive decay measurements. Time-resolved photoluminescence measurements indicate an exponential decay process (or in some cases two exponentials) for bandedge emission with lifetime on the order of 1ns. These samples also exhibit a broad emission in the visible spectral region centered at wavelengths corresponding to green (2.3eV), and demonstrate a non-exponential decay on the order of 100ns-1μs. The temperature dependence of the green band shows strong temperature dependence, with shape changing from a stretched exponential decay at low temperature to power law decay at room temperature. This dramatic change has led us to speculate that there are some intermediate trap states in the material, and that trapping controlled recombination plays an important role in its carrier dynamics. We have examined recombination lifetime in ZnO epilayers via photoconductive decay measurements. Photoconductive decay transients show fast and slow decay components, with the fast time constant less than 500ns (measurement limited) and slow time constant on the order of of milliseconds. The fast decay is representative of the minority carrier lifetime of the ZnO material. The rise and decay portions of the slow photoconductive response were found to persist for several minutes. The photoconductive response is attributed to hole traps in ZnO, where we have used a rate equation model to describe photoconductive characteristics. Based on this model and knowledge of approximate values for hole capture lifetime and carrier recombination lifetime suggested by the time-resolved photoluminescence data, a method was used to extract the hole trap density spectrum. Traps were found to be distributed near 0.7-0.9 eV from the valence band edge, with a sharp dependence on surface passivation with SiO_2. The optical signatures of the defects described in this work will be used to characterize and optimize ZnO materials to achieve reliable p-type doping and high radiative efficiency in ZnO epilayers needed for high-performance UV and visible sources.



(a)



(b)



(c)

Figure 7: (a) time-resolved photoluminescence for band edge and defect-band emission (data courtesy of M. Cheung and A. Cartwright, U-Buffalo) (b) photoconductive decay demonstrating a fast and slow recombination process, and (c) trap density spectrum extracted from slow photoconductive response process

Growth of ZnO on cubic MgO: The most common orientation for ZnO epitaxial growth is the c-plane (0001) orientation, where the polar direction (c-axis) of the wurtzite ZnO crystal is along the growth direction. The epitaxial growth of wurtzite ZnO with the c-axis in the growth plane has also been achieved with several reports of a-plane $(11\bar{2}0)$ ZnO, typically on r-plane sapphire. The growth of ZnO along the non-polar direction is of interest for device applications where polarization and piezoelectric properties at hetero-interfaces are undesirable, or where the polarization and piezoelectric properties are specifically desired in-plane. Furthermore, the ability to integrate the wurtzite phase of ZnO with the rock-salt phase of MgO is of major importance for heterostructures utilizing both wurtzite and rock-salt phases of the MgZnO and CdZnO alloy systems and the possibility for integrating other cubic oxide materials such as perovskite ferroelectrics. We have achieved the growth of non-polar m-plane $(10\bar{1}0)$ ZnO on MgO (001) substrates. ZnO was grown on MgO (001) substrates by plasma-assisted molecular beam epitaxy, and compared to deposition by laser ablation. using a KrF excimer laser, ZnO target, and oxygen ambient. ZnO deposition on MgO by PLD demonstrates a predominant c-axis orientation, with electrical and structural properties similar to the deposition of ZnO on c-plane sapphire for the PLD process in our laboratory. In contrast, ZnO growth by MBE on MgO exhibits a predominant m-plane orientation upon nucleation and subsequent growth, as observed by in situ RHEED. A critical thickness of approximately 0.1μm is observed for m-plane growth, for which ZnO growth beyond this thickness transitions to a c-plane orientation. The m-plane crystalline orientation is further indicated by the observation of x-ray diffraction peaks corresponding to $(10\bar{1}0)$ and $(20\bar{2}0)$ reflections in a θ-2θ scan.

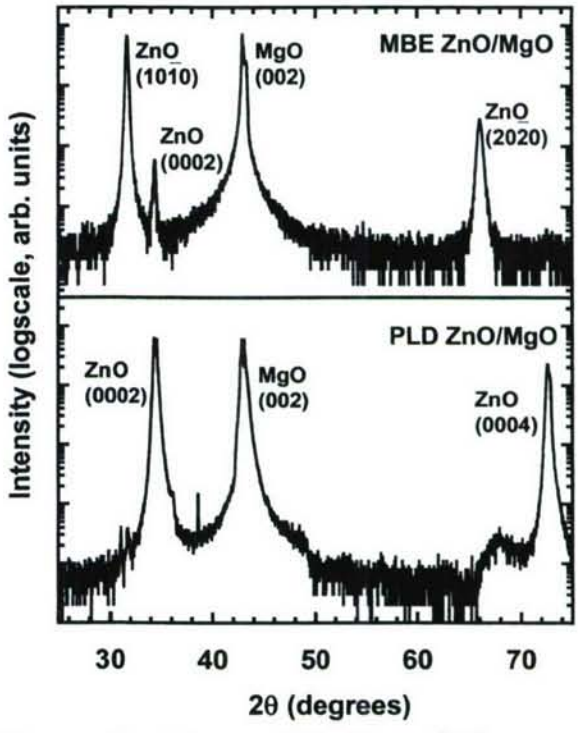


Figure 8: X-ray diffraction θ-2θ scan demonstrating m-plane orientation for MBE growth of ZnO on MgO and c-plane growth for deposition by laser ablation (layer thickness approximately 0.1μm each layer).

ZnO/Ferroelectric Heterostructures: An alternative approach to p-type doping through the intentional introduction of acceptors in ZnO is to use polarization at a heterojunction interface. In this approach, large built-in fields would be used to invert a semiconductor from n-type to p-type material. Furthermore, controlled heterojunctions with highly polar materials provide a unique means of bandgap engineering, which has been exploited for wide bandgap nitride materials. The large polarization associated with ZnO and alloys, along with the potential compatibility of these materials with ferroelectric perovskite oxides, may provide an opportunity to engineer novel devices based on the large and switchable polarization in these materials. A schematic diagram illustrating these ideas are shown in Figure 9(a). We have examined the integration of ferroelectric PZT with ZnO thin films. A memory effect was observed in the capacitance-voltage profile for metal/PZT/ZnO structures, as shown in Figure 9(b). The observed switching behavior is a result of the switchable polarization of the PZT, and is promising for unique

device applications. However, the leakage current is thusfar preventing us from achieving the target goal for this project – the inversion of the ZnO to form a p-type region of material.

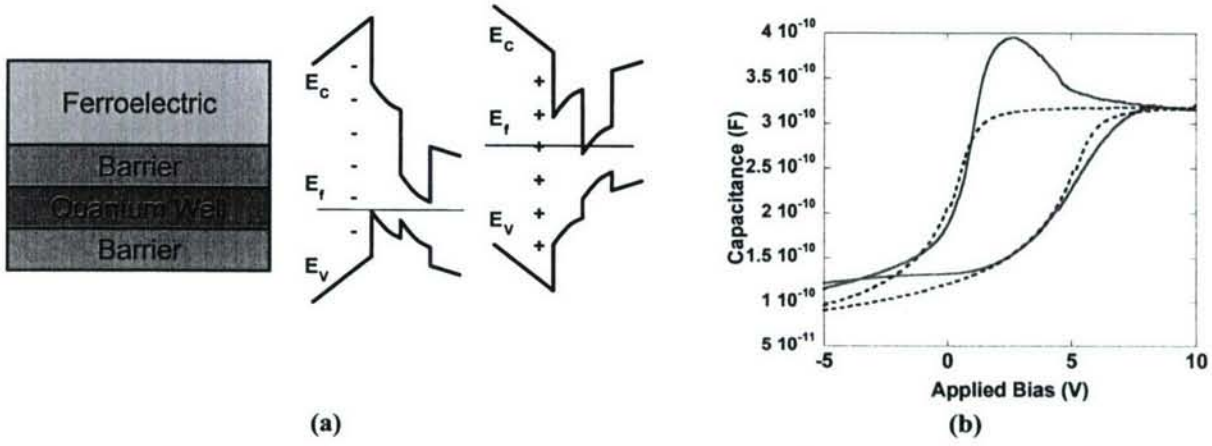
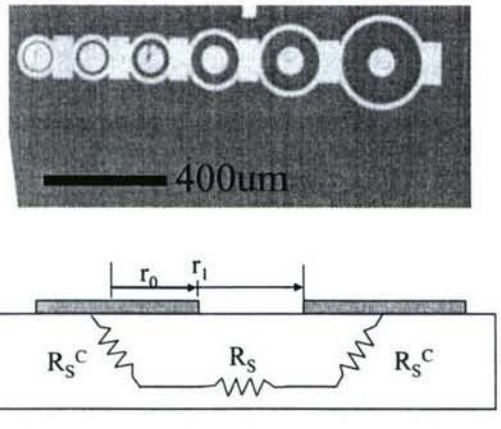


Figure 9: (a) schematic diagram of a metal-ferroelectric-semiconductor structure and corresponding band diagram for two polarization states and (b) capacitance-voltage characteristics for a metal/PZT/ZnO capacitor demonstrating memory due to switchable polarization in PZT.

Ohmic contacts to n-type ZnO with low contact resistivity: The electrical properties of several metal contacts to n-type ZnO (0001) were studied. The ZnO samples consisted of bulk single crystal material, epitaxial layers on sapphire grown by molecular beam epitaxy, and poly-crystalline thin films on sapphire obtained by pulsed laser deposition. Ohmic and rectifying contacts were observed dependent upon both the metal material and the ZnO surface. Ohmic contacts were characterized using the circular transmission line method, where contact resistivity was found to be in the range of 10^{-4}-10^{-5} Ω-cm^2. Schottky behavior was observed using Ag contacts exhibiting varying leakage current and breakdown voltage dependent on the polarity of the ZnO surface. The achievement of ohmic contacts with low contact resistivity is critical for laser diodes. Ohmic contacts are expected to be a major challenge for p-type ZnO. Similar studies will be needed for p-type contacts as reliable p-type ZnO epilayers are developed.



$$R_T = \frac{R_S}{2\pi}\ln\left(\frac{r_1}{r_0}\right) + \frac{R_S^C}{2\pi}\left(\frac{L_T}{r_0}\frac{I_0\left(\frac{r_0}{L_T}\right)}{I_1\left(\frac{r_0}{L_T}\right)} + \frac{L_T}{r_1}\frac{K_0\left(\frac{r_1}{L_T}\right)}{K_1\left(\frac{r_1}{L_T}\right)}\right) \qquad L_T = \sqrt{\frac{\rho_C}{R_S^C}}$$

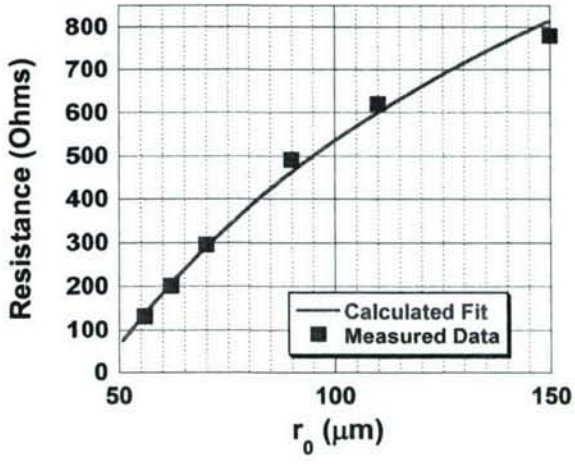


Figure 10: TLM study of n-type contacts on ZnO indicating microscope image of circular TLM test structure, theory behind the measurement, and fitting of I-V data for varying geometry to determine the contact resistivity.

ZnO Thin Film Transistors: The lack of p-n junction devices has inhibited the development of ZnO LEDs and lasers. However, majority carrier devices based on ZnO are clearly possible. We have examined thin film transistors based on ZnO, which show tremendous potential for display applications. The visible transparency, and reasonable electron mobility achievable at low temperature deposition (physical vapor deposition such as sputtering or laser ablation) make this material a superior choice to existing leading technologies such as a-Si or polysilicon. Using ferroelectric thin film insulators, we have achieved high performance ZnO thin film transistors, with representative output characteristics shown in Figure 11(b). The transistor device consists of thin films of BST and ZnO deposited on Pt (gate metal), with source and drain contacts on the surface of the ZnO. The transistor corresponding to the data in Figure 11(b) exhibited an on/off ratio of $I_D^{on}/I_D^{off}=1.5x10^8$, threshold voltage of 1.2V, subthreshold slope of 0.25 V/decade, and minimum off current of 1.8pA. The observed ZnO transistor behavior may also be highly important to the development of reconfigurable systems combining ferroelectric, ferromagnetic, or other multi-functional properties (commonly oxide materials) with semiconductor electronics, due to the inherent compatibility of ZnO to other oxide materials.

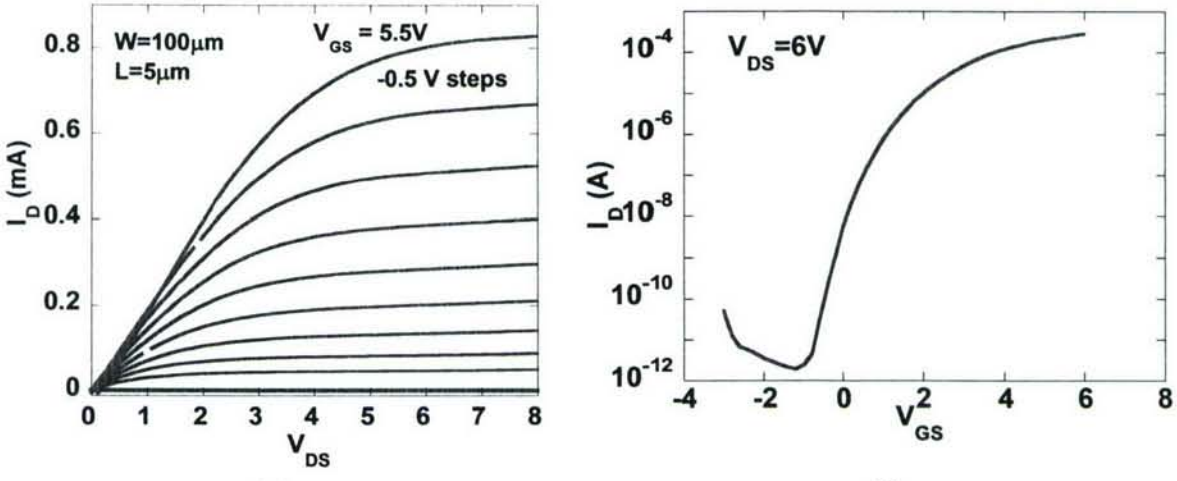


Figure 11: ZnO/BST thin film transistor (a) output characteristics showing transistor action and (b) gate characteristics in saturation showing large I_{on}/I_{off} ratio exceeding 10^8.

PERSONNEL SUPPORTED

A listing of personnel supported by this project are listed below.

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Judi Scramlin-Jones	Administrative assistant
Emine Cagin	Graduate student research assistant
Timothy Murphy	Graduate student research assistant
Weiming Wang	Graduate student research assistant
Jun Yang	Graduate student research assistant

PUBLICATIONS

W. Bowen, W. Wang, E. Cagin and J. D. Phillips, "Quantum confinement and carrier localization effects in $ZnO/Mg_xZn_{1-x}O$ Wells", *Journal of Electronic Materials*, accepted for publication.

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T. E. Murphy, S. Walavalkar and J. D. Phillips, "Epitaxial growth and surface modeling of ZnO on c-plane Al_2O_3", *Applied Physics Letters* **85**, 6338-6340 (2004).

INTERACTIONS/TRANSITIONS

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W. Bowen, W. Wang, E. Cagin and J. Phillips, "Optical and Structural Characterization of $ZnO/Mg_xZn_{1-x}O$ Quantum Wells Synthesized by Pulsed Laser Deposition", Electronic Materials Conference, South Bend, Indiana (2007).

W. Wang, E. Cagin, W. Bowen and J. Phillips, "In-Situ Arsenic Doping of ZnO Grown on GaN/Sapphire and ZnO Substrates by Molecular Beam Epitaxy", Materials Research Society Fall Meeting, Boston, MA (2006).

E. Cagin, W. Wang, J. Yang and J. Phillips, "Epitaxial Growth of m-Plane (10-10) Wurtzite ZnO on Cubic (001) MgO Substrates", 4th International Workshop on ZnO, Giessen, Germany (2006).

E. Cagin, J. Siddiqui, W. Wang and J. Phillips, "ZnO/Ferroelectric Thin Film Heterostructure Capacitors and Thin Film Transistors", 4th International Workshop on ZnO, Giessen, Germany (2006).

J. Phillips, "Semiconducting and ferroelectric oxide materials for optoelectronics", Nano-Optoelectronic Workshop and Summer School of Advances in Photonics, Berkeley, CA (2006).

E. Cagin, J. Yang, J. D. Phillips and P. K. Bhattacharya, "Growth of ZnO on Cubic Substrates by Molecular Beam Epitaxy", Electronic Materials Conference, University Park, PA (2006).

E. Cagin, D. Chen, J. Siddiqui and J. D. Phillips, "Metal-Ferroelectric-Semiconductor Capacitors Based on PZT/ZnO Heterostructures", Electronic Materials Conference, University Park, PA (2006).

J. Siddiqui, E. Cagin, P. Shea, J. Phillips and J. Kanicki, "Incorporation of perovskite oxides into gate insulator of zinc oxide TFTs", Materials Research Society Fall Meeting, Boston, Massachusetts (2005).

J. Phillips, "Epitaxial Growth and Optoelectronic Properties of ZnO", Nano-Optoelectronics Workshop, University of California, Berkeley (2005).

K. Moazzami, T. Murphy and J. Phillips, "Photoconductive Behavior of ZnO for Below Bandedge Excitation", 47th Electronic Materials Conference, Santa Barbara, California (2005).

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E. Cagin, J. Siddiqui, D. Chen, S. Chua, V. Alexander and J. Phillips, "Electrical properties of ferroelectric/ZnO heterojunctions", Materials Research Society Fall Meeting, Boston, Massachusetts (2005).

K. Moazzami, T. Murphy and J. Phillips, "Photoconductive Behavior of ZnO for Below Bandedge Excitation", 47th Electronic Materials Conference, Santa Barbara, California (2005).

P. Emelie, K. Moazzami, J. Phillips, B. Buller and U. Venkateswaran, "Infrared Absorption Characteristics Of Bulk ZnO And Relationship To Electronic Properties", 47th Electronic Materials Conference, Santa Barbara, California (2005).

T. Murphy, K. Moazzami, J. Phillips, M. Cheung and A. N. Cartwright, "Time-Resolved Optoelectronic Properties Of ZnO Epilayers", 3rd International Conference on ZnO and Related Materials, Sendai, Japan (2004).

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T. E. Murphy, D. Chen and J. D. Phillips, "Growth and Electronic Properties of ZnO Epilayers By Plasma-Assisted Molecular Beam Epitaxy", U.S. Workshop on the Physics and Chemistry of II-VI Materials, Chicago, Illinois (2004).

K. Moazzami, T. E. Murphy and J. D. Phillips, "Transient Properties Of ZnO/Al_2O_3 Photoconductors", 2004 Materials Research Society Fall Meeting, Boston, Massachussetts (2004).

NEW DISCOVERIES, INVETIONS, OR PATENT DISCLOSURES

None to report.

HONORS/AWARDS

P. Bhattacharya

- Elected Fellow of the American Physical Society
- IEEE (NTC) Nanotechnology Pioneer Award
- TMS John Bardeen Award

J. Phillips

- DARPA/MTO Young Faculty Award